

Claims

- [c1] 1. A controlled-flow microwave instrument for chemical synthesis that includes heterogeneous or highly viscous materials, said instrument comprising
- a microwave source for generating electromagnetic radiation in the microwave frequencies;
 - a microwave cavity in wave communication with said source for exposing compositions placed therein to microwave radiation;
 - a microwave-transparent pressure resistant reaction vessel in said cavity;
 - a source reservoir for starting materials and related compositions;
 - a pump in communication with said source reservoir for pumping heterogeneous or highly viscous materials from said source to said reaction vessel; and
 - a pressure-resistant valve between said pump and said reaction vessel for isolating said reaction vessel from said pump and said source during application of microwave energy to compositions in said vessel and from any resulting high pressures generated therein.
- [c2] 2. An instrument according to Claim 1 wherein said cav-

ity is selected from the group consisting of single mode and dual mode cavities.

- [c3] 3. An instrument according to Claim 1 wherein said pump comprises a peristaltic pump.
- [c4] 4. An instrument according to Claim 1 wherein said pump is selected from the group consisting of diaphragm pumps and pneumatic pumps.
- [c5] 5. An instrument according to Claim 1 comprising a temperature detector associated with said cavity for measuring the temperature of items in said cavity.
- [c6] 6. An instrument according to Claim 4 comprising means for adjusting the microwave power applied from said source to said cavity.
- [c7] 7. An instrument according to Claim 6 comprising a processor in operative communication with said temperature detector and said adjusting means for adjusting the microwaves applied from the source to the cavity in response to the temperature measured by said temperature detector.
- [c8] 8. An instrument according to Claim 7 comprising means for cooling said vessel in said cavity.
- [c9] 9. An instrument according to Claim 7 comprising means

for cooling said vessel in said cavity during the application of microwaves.

- [c10] 10. An instrument according to Claim 8 wherein said cooling means is in operative communication with said processor for operating said cooling means in response to the temperature measured by said temperature detector.
- [c11] 11. An instrument according to Claim 1 wherein said source is selected from the group consisting of magnetrons, klystrons and solid state devices.
- [c12] 12. An instrument according to Claim 1 wherein said pressure-resistant valve comprises a ball valve.
- [c13] 13. An instrument according to Claim 1 wherein said ball valve is pressure resistant to at least 175 psi.
- [c14] 14. An instrument according to Claim 1 wherein said ball valve is pressure resistant to at least 250 psi.
- [c15] 15. An instrument according to Claim 1 wherein said pressure-resistant reaction vessel has sufficient strength to maintain reactions therein at pressures of at least about 175 pounds per square inch.
- [c16] 16. An instrument according to Claim 1 wherein said pressure-resistant reaction vessel has sufficient strength

to maintain reactions therein at pressures of at least about 250 pounds per square inch.

[c17] 17. An instrument according to Claim 1 wherein said pump is a two-way pump for adding materials to and pumping materials from said reaction vessel, and said instrument further comprising:

- a multi-port valve between said source reservoir and said pump for controlling the flow of materials to and from said reaction vessel;
- a second source reservoir for starting materials OLE_LINK1 in fluid communication with said multi-port valve OLE_LINK1 for providing said reaction vessel with a second set of starting materials;
- a product reservoir in fluid communication with said multi-port valve for collecting reaction products from said reaction vessel; and
- a processor in operative communication with said two-way pump and said multi-port valve for directing the flow of materials to and from said multi-port valve, said two way pump, and said reaction cell.

[c18] 18. An instrument according to Claim 17 and further comprising:

- a plurality of fluid lines for proving fluid communication within said instrument;
- a solvent supply in fluid communication with said multi-

port valve for providing solvent to said reaction cell and for rinsing and cleaning said fluid lines;
a vent in fluid communication with said multi-port valve for venting gases from said reaction vessel;
a waste reservoir in fluid communication with said multi-port valve for receiving waste product; and
a gas supply in fluid communication with said multi-port valve and with said fluid lines for supplying gas pressure to urge materials through said fluid lines.

- [c19] 19. A method of conducting microwave assisted chemical reactions using high viscosity liquids or heterogeneous mixtures of liquids and solids, the method comprising:
- pumping a discrete portion of a composition selected from the group consisting of high viscosity liquids and heterogeneous mixtures of liquids and solids to a microwave-transparent pressure resistant reaction vessel at ambient pressures of between about atmospheric pressure and about 30 psi;
 - isolating the discrete portion in the pressure resistant vessel;
 - applying microwave radiation to the isolated discrete portion in the reaction vessel to initiate and maintain a chemical reaction at a pressure of at least about 175 psi while preventing the vessel from releasing higher-

pressure gases generated by a chemical reaction in the vessel;

releasing pressure from the vessel following desired completion of the chemical reaction; and

pumping the reaction products of the discrete portion from the vessel at ambient pressures of between about atmospheric pressure and about 30 psi following the pressure release.

[c20] 20. A method according to Claim 19 comprising pumping a second discrete portion after the first has been pumped out and thereafter carrying out the steps of isolating the second portion, applying microwave radiation to the second portion, releasing pressure from the vessel, and pumping reaction products from the vessel.

[c21] 21. A method according to Claim 19 comprising stirring the discrete portion in the reaction vessel during the step of applying microwave radiation.

[c22] 22. A method according to Claim 19 wherein the step of applying microwave radiation comprises applying single mode radiation.

[c23] 23. A method according to Claim 19 wherein the step of applying microwave radiation comprises applying dual mode radiation.

- [c24] 24. A method according to Claim 19 comprising maintaining the reaction vessel at a constant temperature for extended portions of a chemical reaction.
- [c25] 25. A method according to Claim 19 comprising measuring the temperature of the reaction vessel and adjusting the application of microwave radiation in response to the measured temperature.
- [c26] 26. A method according to Claim 19 comprising measuring the temperature of the reaction vessel and cooling the reaction vessel in response to the measured temperature.
- [c27] 27. A method according to Claim 19 comprising maintaining the pressure in the reaction vessel at between about 175 and 250 psi.
- [c28] 28. A method according to Claim 20 comprising rinsing the reaction vessel with a solvent between the steps of pumping the reaction products and pumping the second discrete portion.
- [c29] 29. A method according to Claim 28 comprising driving the rinsing solvent with an inert gas.
- [c30] 30. A method according to Claim 19 comprising mixing the composition from components selected from the

group consisting of solids, liquids, solutions, solid phase catalysts and solid-supported reagents prior to the step of pumping the composition to the reaction vessel.